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The influence of displacement damage on deuterium retention in tungsten exposed to divertor plasma in DIII-D*

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Abstract

Experiments were conducted to determine the influence of displacement damage on retention of deuterium in tungsten plasma-facing components in a tokamak. Tungsten samples, previously damaged by ion irradiation, were exposed to the outer strike point of attached H-mode plasmas in DIII-D. Nuclear reaction analysis (NRA) was used to measure the depth profile of deuterium retained in the tungsten. Displacement damage increased the concentration of retained deuterium to the maximum depth (about 2.5 microns) of the damage, to concentrations up to 0.003 D/W, compared to D/W < 10^{-5} in undamaged W. Tungsten coverage on adjacent carbon surfaces of the probe was mapped by Rutherford backscattering, giving the average tungsten erosion rate and spatial variation of redeposition.

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1. Introduction

Neutron or ion irradiation cause atomic collision cascades creating lattice defects that can increase retention of hydrogen isotopes in metals exposed to plasmas. This increased retention could potentially impact tritium inventory in ITER where tungsten plasma-facing components will be damaged by fusion neutron irradiation [1]. Increased retention of deuterium at displacement damage has been studied in laboratory experiments in which tungsten samples damaged by ion irradiation were exposed to deuterium (D) plasmas or low-energy D ion implantation [1,2, 3]. Here we describe an experiment to determine whether displacement damage also increases deuterium retention in tungsten exposed to fusion plasmas in a tokamak divertor environment. Laboratory studies provide more controlled conditions during plasma exposure, which is needed to elucidate physical mechanisms. However, the goal of these studies is to predict plasma-wall interactions in the more complex tokamak environment, so it is also necessary to conduct tests in tokamaks to detetermine whether similar behavior is observed.

The experiments described here are similar to a previous study [1] in which tungsten was exposed in the PISCES linear plasma device, except that this time the samples were exposed to divertor plasma in DIII-D. The samples were tungsten samples with displacement damage before exposure up to about 0.6 displacements per atom (dpa). The results show that displacement damage also increases the deuterium retention in tungsten exposed to tokamak divertor plasma, even though the exposure times are relatively short compared to the previous laboratory studies.

Deposition of tungsten onto adjacent graphite surfaces is examined by Rutherford backscattering (RBS). The results show tungsten eroded from the samples by the plasma is

redeposited within a few millimeters. The amount of deposited tungsten indicates an average net tungsten erosion rate of 0.16 nm/sec, in agreement with previous measurements [4].

2. Experimental Methods

Samples were prepared from Plansee tungsten (99.97% pure). Tungsten disks 6 mm in diameter and 1.6 mm thick, were polished and vacuum annealed at 1000ºC for one hour to remove surface damage from polishing. The samples were then damaged by irradiation with 12 MeV silicon ions at room temperature to fluences of $5x10^{13}$ and $5x10^{14}$ ions/cm^2. Damage was calculated using the binary collision particle transport code SRIM-2006 [5] using a displacement threshold energy of 40 eV [1]. The damage peaks near the end-of-range, about 2 microns beneath the surface, at 0.06 and 0.6 dpa respectively for the two fluences. Since the number of displacements per ion is large (~7500), the concentration of implanted silicon is small and is comparable to that of residual impurities and therefore should have little influence on D retention.

This study utilized the Divertor Material Exposure System (DiMES) [6] to expose tungsten samples to the divertor plasma in DIII-D. Samples were exposed during 10 medium-density ELMing H-mode lower single null deuterium discharges with attached plasma at the outer divertor. The outer strike point was moved onto the DiMES probe for about 3.5 seconds during the high power portion of each shot, giving a total exposure time of 35 seconds. The samples were coplanar with the graphite probe face and adjacent lower divertor tiles. Six tungsten samples were exposed in the configuration shown in figure 1. The two samples at positions A and B had the higher (0.6 dpa peak) displacement damage. The two samples at positions D and E had the lower (0.06 dpa peak) damage. For comparison, an undamaged Plansee tungsten sample was exposed at the center position G. Sample position F

was occupied by a vacuum plasma sprayed tungsen sample for another study and position C was occupied by a graphite button during the exposure.

An infrared TV (IRTV) diagnostic measured the surface temperature of graphite divertor tiles versus time and space during the discharges. The surface temperature at the major radius of DiMES increased from about 60°C to 350°C. From the temperature rise, and known thermal properties of graphite, the absorbed heat flux was calculated. The heat flux had a peak value of 170±30 W/cm^2 and a width of 3.5±1 cm FWHM. The major radius of the peak heat flux was 148.4±0.5 cm, which is centered on the DiMES probe, for about 3.5 seconds during the high power portion of each discharge. A thermocouple on the DiMES probe showed a temperature rise of 20 to 30°C 20 seconds after a shot, which is roughly consistent with the above heat flux and the heat capacity of the probe. The probe temperature just before each shot increased from 26°C to 66°C during the sequence of 10 shots.

After exposure to the DIII-D plasma, the deuterium depth profile within 3 microns of the surface was measured using D(^{3}He,p)α nuclear reaction analysis (NRA) with an analysis beam of 2.5 MeV ^{3}He ions. Energy spectra (yield versus energy) of the protons were measured using a silicon surface barrier detector. A large-area (300 mm^2) detector close to the sample was used to obtain high sensitivity. A large depletion depth (1500 μm) is necessary to stop the energetic protons. An annular detector geometry was used to minimize kinematic broadening of the energy resolution. A 12 μm thick mylar foil is used to stop elastically scattered ^{3}He from reaching the detector. Protons originating deeper beneath the surface reach the detector with higher energy than those from nearer the surface. Thus the energy scale can be converted to a depth scale. This was done using the SIMNRA simulation program [7]. SIMNRA was also used to convert the measured yield to a concentration. This was done using a simulated spectrum for a uniform concentration D/W=0.001, small enough

not to significantly influence the stopping power. The measured concentration was determined from the ratio of the measured to simulated yields times the concentration used in the simulation. The areal density of carbon on the surface of the tungsten samples was also determined from the yield of protons from the $^{12}C(^{3}He,p)^{14}N$ nuclear reaction. These protons produce peaks in the same spectra used for D profiling, but at lower energy than the protons from the $D(^{3}He,p)\alpha$ reaction.

Another purpose of this study was to examine erosion and redeposition of tungsten. This was done by mapping the coverage of tungsten on the 5 cm diameter graphite face of the DiMES probe using Rutherford backscattering (RBS) with 2 MeV ^{4}He..

3. Results and discussion

3.1 Tungsten deposition and erosion

Erosion and deposition of tungsten were also examined in this experiment. Rutherford backscattering of 2 MeV ^{4}He was used to measure the tungsten coverage on the face of the graphite sample holder. While the erosion of tungsten could not be measured directly from any change in the tungsten samples, an average erosion rate could be inferred from the measured tungsten deposition onto the adjacent graphite. Furthermore, the measurements show the spatial distribution of the deposited tungsten. The results show that the eroded tungsten is mainly redeposited within a few millimeters of the source. Prior to the experiment, the probe face was cleaned and determined (by RBS) to be free of tungsten, to insure that the tungsten measured after the exposure in DIII-D was all from this experiment. Examination by optical microscopy showed no indications of arcing on the samples. The mechanism of erosion is probably physical sputtering by energetic ions from the plasma.

Figure 1 shows a map of tungsten coverage on the probe cap. Measurements were made at 155 locations indicated by the small circles. A contour map of coverage was obtained by

interpolation between measured locations. The tungsten coverage varied from about $2x10^{16}$ atoms/cm^2 near the tungsten samples to about $2x10^{14}$ atoms/cm^2 near the edge of the probe. The tungsten coverage decreased towards the edges of the probe with a characteristic e-folding length of about 3 mm. This shows that the characteristic distance tungsten was transported across the surface by the plasma during the exposure was similar to the tungsten button dimensions but small compared to the DiMES probe size. Integrating the tungsten coverage over area gives $6\pm1 \times 10^{16}$ atoms for the amount deposited onto the graphite. Assuming that all of the eroded tungsten was redeposited onto the DiMES probe, as seems likely from the sharp decrease in coverage with distance from the source, the tungsten erosion averaged over the area of the six samples is $3.5x10^{16}$ atoms/cm^2 or 5.6 nm, and the erosion rate, averaged over the 35 second exposure, is 0.16 nm/sec. The erosion rate and e-folding length for redeposition determined here are similar to values determined in a previous experiment in DIII-D [4].

3.2 Deuterium retention

Figure 2 shows the deuterium concentration versus depth in the 5 Plansee tungsten samples. Table 1 lists the deuterium concentrations at the depth of peak damage (2 microns), and areal densities of deuterium obtained by integrating the concentration to a depth of 3 microns. Beneath the surface, ie at depths beyond 0.3 micron, the D concentration is below the detection limit of about 10 appm in the undamaged sample but much higher in all of the damaged samples. Examination by optical microscopy showed no evidence of blisters on the sample surfaces. This is consistent with the low sub-surface D retention in the undamaged tungsten. In the damaged samples, the D concentration extends throughout the damaged region, but decreases abruptly beyond 2.3 microns where the damage decreases. The

displacement damage clearly has increased the D concentration, with maximum values of about 0.00012, 0.00038, 0.0009 and 0.0027 D/W for samples A,D,E, and B respectively, at the depth of maximum damage. However, the D concentration does not vary consistently with the amount of damage from one sample to another. For example, samples A and B both had the higher damage, but the D concentration was about 30 times less in sample A than in sample B. Similarly, samples D and E both had the lower damage but the D concentration was about 3 times less in sample D than in sample E. This difference in D retention for nominally similar conditions indicates an uncontrolled condition during exposures. The most likely parameter is the sample temperature. The temperature increase of a sample during plasma exposure will depend on the heat flux and the thermal contact between the sample and the graphite sample holder. The heat flux varies with major radius, but all the samples should have been within the FWHM of the peak heat flux according to information from the IRTV. Furthermore, the heat flux is expected to be uniform along the toroidal direction. The fact that the sample with the lower D was on the inboard side for samples A and B but on the outboard side for samples D and E is evidence that the variation in D retention is not due to variations in heat or particle flux.

A more likely explanation is that the thermal conductance to the holder varied from sample to sample. The IRTV showed that the heat flux was sufficient to raise the surface temperature of a graphite tile by about 300°C during a shot. If the thermal contact between samples and holder is good, the temperature of a sample should be close to the temperature of the holder and should therefore rise by a similar amount. If a tungsten sample is thermally isolated from the holder, a heat flux of 170 W/cm^2 for 3.5 seconds should increase its temperature by 1150°C. Retention of D at damage in tungsten depends strongly on the temperature of the sample during exposure to the plasma, and is very low at temperatures of

500°C and higher [1]. It is therefore possible that D retention was influenced by heating of the samples by the plasma, which could have differed between the samples. Figure 3 shows the deuterium retention in tungsten samples damaged by ion irradiation to a peak damage level of 0.6 dpa, then exposed in PISCES at 200°C to D plasma [1], and at 300°C and 420°C to D+5% He plasma. There is a large decrease in D retention between 300°C and 420°C. This decrease in D retention at higher temperatures appears to be due to a combination of defect annealing and thermal detrapping. The D retention observed in the samples exposed in DIII-D is between the retention observed in samples exposed in PISCES at 200°C and at 420°C , and is therefore consistent with sample temperatures during plasma exposure in this range which seem to be possible given the high heat flux at the OSP.

The possibility that D uptake into the tungsten might have been influenced by deposition of carbon on the tungsten was also considered. However, the outer strike point of attached plasmas is generally a region of net erosion rather than deposition [8]. In this experiment, the presence of tungsten on the graphite probe face indicated net erosion of the tungsten, as discussed below. Furthermore, the carbon coverage on the tungsten was measured by NRA. Variations in carbon coverage were not large (see table 1). Also, the samples with low D concentrations had less carbon than those with more D, indicating that D uptake was not inhibited by surface carbon. The D depth profiles (figure 2) show that D retention in the damaged samples was beneath the surface and therefore not primarily in the carbon on the surface.

4. Conclusions

Erosion and deuterium retention were examined in tungsten samples exposed to the outer strikepoint of attached ELMing H-mode plasmas in DIII-D. An average net tungsten

erosion rate of 0.16 nm/sec, and a characteristic transport distance of about 3 mm, were determined from measurements of tungsten deposition onto the adjacent surface of the graphite sample holder.

Displacement damage increased the concentration of retained deuterium to the maximum depth (about 2.5 microns) of the damage, to concentrations up to 0.003 D/W. Previous studies have shown that damage increases D retention in tungsten exposed to linear plasma devices or ion implantation, but this study shows for the first time that similar increases also occur in plasma-facing components in a tokamak. Although D retention in this study varied, possibly due to variable sample temperature, the higher values are similar to values seen previously in similar samples exposed to plasma in the PISCES linear plasma source, even though the duration of exposure was about two orders of magnitude shorter in DIII-D. This study demonstrates that displacement damage can increase D retention in tungsten plasma-facing components up to a few tenths of an atomic percent. The impact this will have on tritium inventory in tritium fuelled machines such as ITER will strongly depend on the depth to which retention extends. This depth appears to be small at low temperatures but to increase at higher temperature [1]. The temperature of the tungsten also strongly affects the concentration of D retained at damage, being low above 500°C but higher below 300°C. Thus, the effect of damage on tritium inventory might be small at temperatures sufficiently low that tritium uptake is kinetically limited, and also low at temperatures high enough that it is not bound to the traps, but there could be an intermediate temperature range, around 300°C, where the permeation is fast enough and the trapping is still strong enough to result in significant concentrations to depths large enough to impact tritium inventory.

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Sample	Dpa at damage peak	D concentration at damage peak (10^{-3} D/W)	D within 3 μm (10^{16} atoms/cm^2)	Surface carbon (10^{16} atoms/cm^2)
A	0.6	0.12	0.11	2.0
B	0.6	2.7	3.5	4.2
D	0.06	0.38	0.3	3.5
E	0.06	0.9	1.3	3.8
G	0	< 0.001	0.08	5.1

Table 1

The damage levels and D concentrations at the depth of peak damage (2 microns), the areal density of D within 3 microns of the surface, and coverage of surface carbon for each tungsten sample.

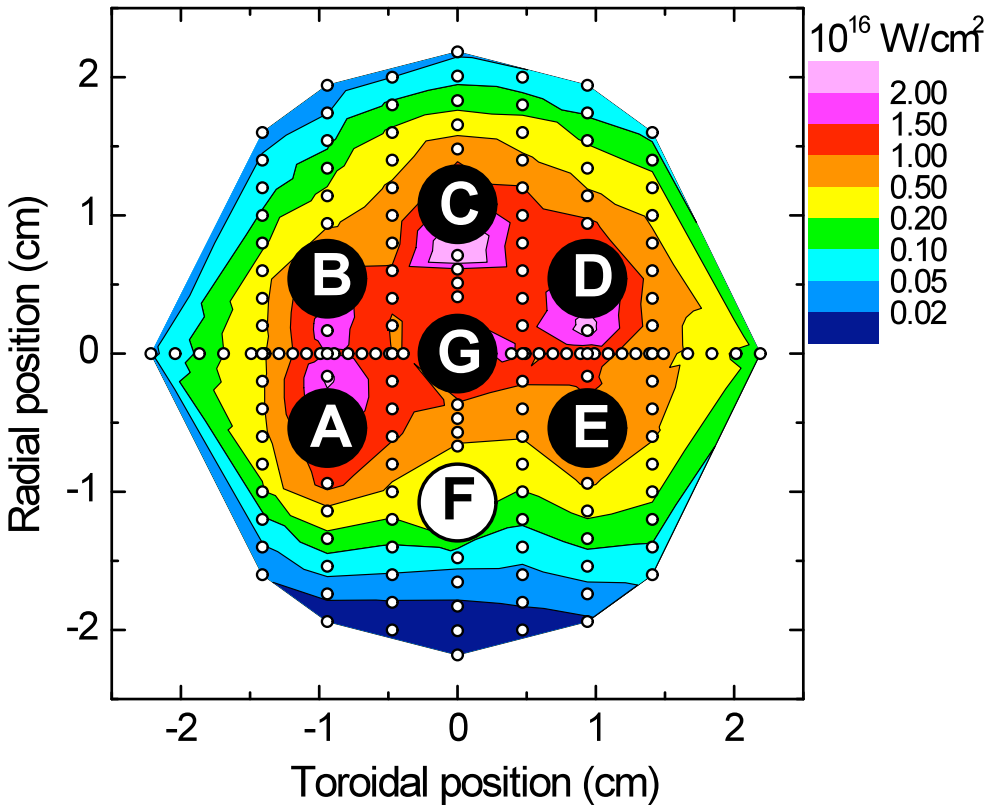

Figure 1 Tungsten coverage on the graphite DiMES probe face. The small white dots indicate the 155 locations where RBS measurements were made. The large black dots indicate the positions (A to G) of tungsten samples. The white sample position (F) was occupied by graphite during the plasma exposure. Vertical and horizontal coordinates are along the radial and toroidal directions in DIII-D.

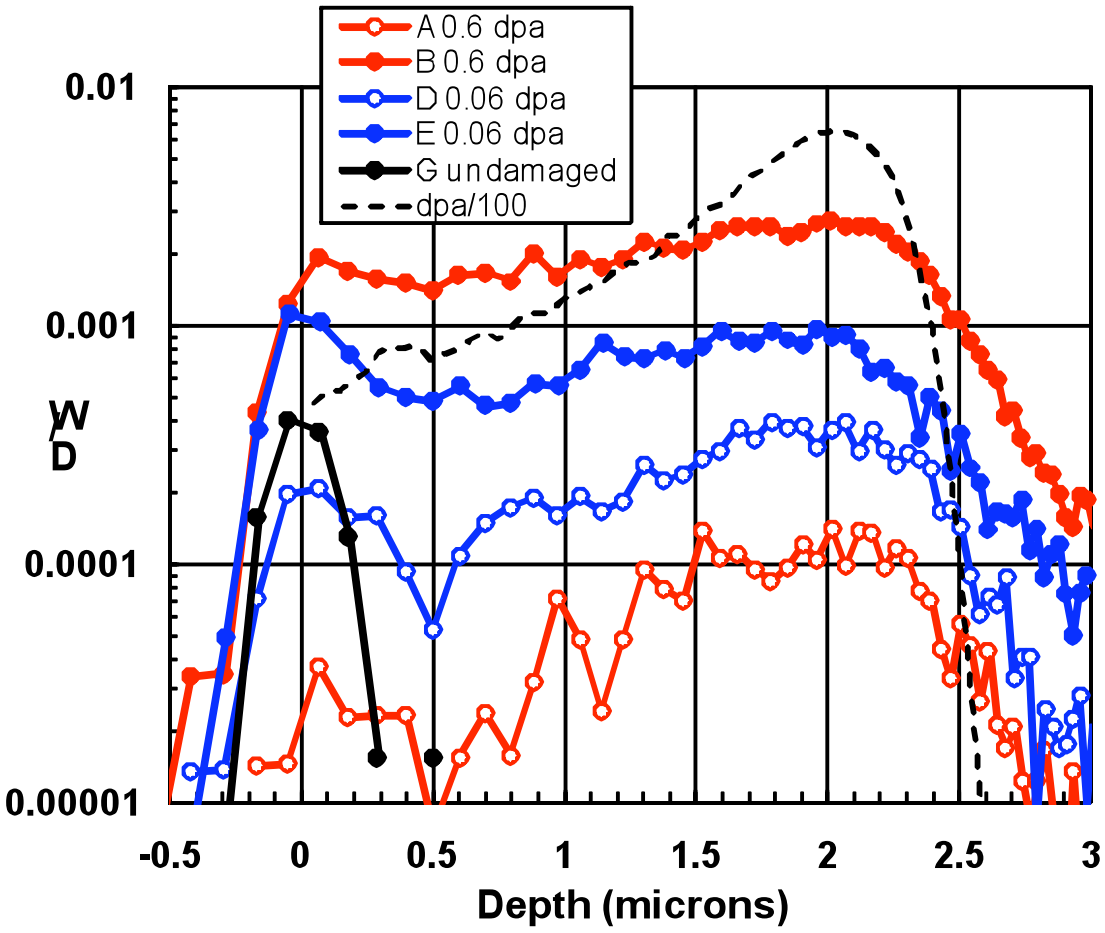

Figure 2 Deuterium concentration versus depth in the 5 Plansee tungsten samples exposed in DIII-D. The black dashed line shows the calculated displacement damage for the 0.6 dpa peak samples. Finite values of concentration at negative depth are due to the finite depth resolution of the measurement technique.

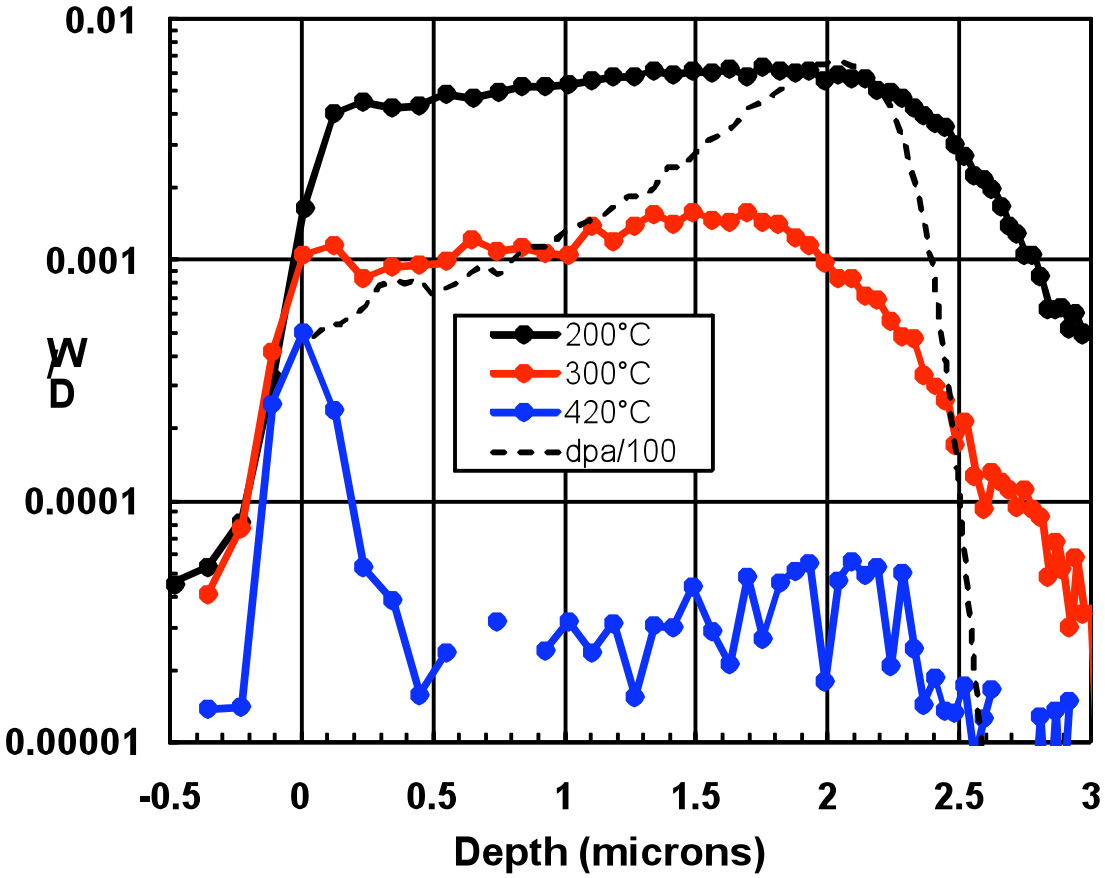

Figure 3 Deuterium concentration versus depth in tungsten samples damaged by ion irradiation to a peak damage levelof 0.6 dpa, then exposed in PISCES at 200°C to D plasma (black), and at 300°C (red) and 420°C (blue) to D+5% He plasma.

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